

Multi-scale Directed Surface Topography Machined by Electro Discharge Machining in Combination with Plasma Electrolytic Conversion for Improved Osseointegration

Fritz Klocke, Max Schwade*, David Welling

Laboratory for machine tools and production engineering (WZL)

RWTH Aachen University

Steinbachstr. 19, 52074 Aachen, Germany

f.klocke@wzl.rwth-aachen.de

m.schwade@wzl.rwth-aachen.de

d.welling@wzl.rwth-aachen.de

*Corresponding author

Alexander Kopp

Meotec GmbH & Co. KG

Pauwelsstr. 17, 52074 Aachen, Germany

alexander.kopp@meotec.eu

Abstract: Major deficits concerning biodegradable and non-biodegradable orthopaedic implants are a result of insufficient osseointegration and an accelerated corrosion rate. These in turn are significantly influenced by the surface tissue-interface. Cells belonging to different steps of the osteoblast differentiation cascade respond differently to the same surface structure. Therefore, a combination of particular micro and macro structures resulting in a multi-scale directed surface topography may significantly improve the cell response throughout the whole osseointegration process. This paper proposes the possibility to adapt the structural properties of a surface independently throughout different magnitudes of topography by a combination of Electro Discharge Machining and a Plasma Electrolytic Conversion process. Examples of such structures are demonstrated in samples made of the magnesium alloy WE43 as well as the most commonly used titanium alloy for orthopaedic implants Ti6Al4V.

Keywords: EDM, Wire-EDM, Sinking-EDM, PEC

Biographical notes: Fritz Klocke: 1950: Born October 10 in Vlotho; 1965 - 1968: Apprenticeship as toolmaker; 1970 - 1973: Production engineering studies at Lemgo / Lippe Polytechnic; 1973 - 1976: Production engineering studies at Technical University Berlin; 1976 - 1981: Assistant at the Institute for Machine Tools and Production Engineering, Technical

F. Klocke et al.

University Berlin; 01.09.1981: Chief Engineer; 1982: Doctorate in engineering; 1984 - 1994: Employed at Ernst Winter & Sohn GmbH & Co., Norderstedt; 01.10.1984: Head of Process Monitoring; 01.09.1985: Technical Director, Mechanical Engineering Department; 1985: Award of Otto Kienzle Medal by the Universities Production Engineering Group; since 1995: Director of the Chair of Manufacturing Technology at the Institute for Machine Tools and Production Engineering (WZL) of the RWTH Aachen and director of the Fraunhofer Institute for Production Technology, Aachen. 2001 - 2002 Dean of the Faculty for Mechanical Engineering. 2006: Honorary Ph.D. by the University of Hannover. 2007 – 2008 President of the International Academy for Production Engineering (CIRP); 2009 Honorary Ph.D. by the University of Thessaloniki; 2010 Honorary Ph.D. by the Keio University; 2010 Award of Fraunhofer Medal; Married to Prof. Dr.-Ing. Martina Klocke, 3 children;

Max Schwade received his diploma in electrical engineering from the TU Dortmund in 2008. Since 2009 he is a research assistant at the chair of manufacturing technology at the laboratory for machine tools and production engineering (WZL) of RWTH Aachen University. His research focuses on the machining of biomaterials by EDM and the influence of this process on the biocompatibility.

David Welling is a research assistant since February 2010 in the group of EDM/ECM at the chair of manufacturing technology of the laboratory for machine tools and production engineering (WZL) of the RWTH Aachen University. His research activities focus on the manufacturing technology Wire-EDM, especially in field of conventionally hard to machine materials for turbine engine applications. Additionally he coordinates the technical work for the EU-funded project ADMAP-GAS.

Alexander Kopp studied production engineering at the RWTH Aachen University. After finishing his Diploma with honors he founded his own company, Meotec. Already during his thesis at Continental he focused on electro chemical surface treatments for light metals. Later he used his experience in this field to develop and optimize electro-chemical surface treatments for medical implants (with Meotec).

Introduction

Implants made of metallic biomaterials have a wide range of applications. Due to their superior mechanical properties, they are especially suited for orthopaedic treatments (Niinomi 2002). One example is the replacement of missing bone after a tumor extraction or a severe fracture. The most well-known application is the replacement of missing joints like hip and knee as well as temporary implants like fixation screws and fracture plates. Population growth on the one hand, over ageing of society and unhealthy nutrition in the industrial countries on the other hand result in a rapid growth of medical devices industry, especially in the US, Europe and Japan. Expenses exceeded \$220 billion in the U.S. national health care system in 2008 and are predicted to experience continued robust growth at a compound annual growth rate of 12% (Medical Manufacturing 2009). Several million people are treated with metallic implants every year. This shows the relevance to optimize the performance of applied implants in order to increase the quality of life for sufferers of bone issues. Most common metallic materials used for permanent implants are titanium, stainless steel or cobalt-chromium alloys due to their superior mechanical properties and biocompatibility (Niinomi 2002). Especially, titanium is known for its longevity and low complication rate due to the high corrosion resistance. In addition to this, virtually no known allergies are related to titanium. But even low complication rates in the magnitude of 1 - 2% result in several thousands of patients suffering from severe inflammatory reactions which lead to serious pain and complex revision surgeries. It is estimated that approximately 1 million hip replacements and 250,000 knee replacements are carried out per year (Nasab 2009). Typical problems resulting from titanium implants are stress shielding and the loosening of the implant seat as a result of insufficient osseointegration. This is especially the case for low quality bone structure as it is usually found in elderly or weakened patients after certain therapies, e.g. chemotherapy. Other observed complications are inflammatory reactions which are often a result of foreign material loaded on the implant surface by the manufacturing chain or bacterial contaminations caused during insertion. But even without any complication, the longevity of titanium implants is usually limited to 10-15 years despite the good fatigue strength of titanium. Therefore revision surgeries are necessary for temporary implants as well as permanent implants leading to an increased complication rate, longer recreation times and higher health care costs. Despite the good biocompatibility of titanium

and other metallic biomaterials, the performance of today's implants is still in need of improvement.

To overcome the above mentioned drawbacks of state of the art implants, biodegradable materials are in the focus of recent research approaches in the medical engineering sector. Especially, magnesium is one of the most promising materials for orthopaedic applications (Staiger et al. 2006 and Witte et al. 2009). Magnesium is able to provide the needed structural stability compared to most other biodegradable materials, i.e. biodegradable polymers. In fact, the mechanical properties are very similar to those of the human bone and the application of magnesium implants may therefore prevent stress shielding, as seen in table 1.

Table 1: Excerpt of the mechanical properties of titanium and magnesium compared to those of cortical bone (Staiger et al. 2006)

Properties	Density ρ / (g / cm ³)	Elastic modulus E / GPa	Compressive yield strength σ / MPa	Fracture toughness K / (MPam ^{1/2})
Natural bone	1.8 - 2.1	3 - 20	130 - 180	3 - 6
Titanium	4.4 - 4.5	110 - 117	758 - 1117	55 - 115
Magnesium	1.74 - 2.0	41 - 45	65 - 100	15 - 40

In contrast to implants made of titanium, the degradation of magnesium implants caused by corrosion is not solely a tolerated side-effect, but a desired feature. The purpose of the implant in this case is to provide the structural stability of the replaced bone tissue until the natural recreation abilities can fix the gap on their own and the ingrowing bone regains initial strength. Therefore degradation must be modified in such a way, that the corrosion rate progresses in the same speed range as the ingrowth of bone occurs (Denkena and Lucas 2007). To keep contact with the adjacent bone, the decreasing volume of the implant always has to provide a sufficiently small gap at the implant-tissue interface throughout the whole healing process.

The most dominant advantages of biodegradable implants are the prevention of revision surgeries and the avoidance of foreign material residues within the human body. Nevertheless several technical barriers still prevent the clinical application of biodegradable magnesium implants. These are predominantly the accelerated degradation behaviour and the spontaneous release of high amounts of hydrogen gas shortly after insertion (Gu and Zheng 2010). As a result of the early resorption, the

initial structural stability of the implant is lost too fast and the ingrowing bone is unable to stabilise sufficiently. The gas release increases the pressure in the surrounding area causing painful gas blisters and preventing the necessary ingrowth of new healthy bone. Furthermore, the accumulation of hydrogen leads to a pH-shift causing irritations within the adjacent tissue and an increase of the rejection risk of the implant.

Resulting from these deficits, the performance of orthopaedic implants made of biodegradable as well as non-biodegradable materials need to be improved. According to the definition of D.F. Williams (Williams 2008) these deficits can be summarized as lacking biocompatibility: "Biocompatibility refers to the ability of a biomaterial to perform its desired function with respect to a medical therapy, without eliciting any undesirable local or systematic effects in the recipient or beneficial of the therapy, but generating the most appropriate beneficial cellular or tissue response in the specific situation, and optimising the clinically relevant performance of that therapy". In terms of orthopaedic bone implants the function of the lost bone must be substituted as best as possible avoiding any negative effect to the surrounding tissue and ensuring biofunctionality.

To improve the biocompatibility of biodegradable and non-biodegradable implants the osseointegration and corrosion rate should be regarded as key factors. By understanding how to regulate these two aspects, the lifetime of both implant groups can be raised and negative influences on the surrounding tissue minimized. Regarding implants made of titanium the loosening - a typical cause of revisions surgeries - could be prevented. Concerning magnesium implants the corrosion rate can be matched with the growth rate of the recovering bone, i.e. decelerating the corrosion and accelerating the growth rate of bone.

For both aspects the implant and tissue interface is most important and therefore functional surfaces tailored to the characteristics of the therapy are required (Kiesewetter et al. 1996). In this paper an approach is presented to improve the cell adhesion and corrosion resistance of implants made of titanium and magnesium by a unique micro and macro structure machined by Electro Discharge Machining in combination with a Plasma Electrolytic Conversion process. For demonstration purposes machining tests were performed in the titanium alloy Ti6Al4V and the magnesium alloy WE43.

Implant-Tissue Interface

The biocompatibility and therefore the functionality and biological compatibility of an implant is influenced by the material itself, e.g. the composition of the used alloy. The main aspect, if not the most important, is the implant and tissue interface and therefore the surface properties. These properties are influenced by the base material, the machining process and an optional surface treatment. Therefore any negative impact on the biological compatibility resulting from the machining process should be avoided. Instead, the implant surface has to be functionalized by properties that improve the therapy. To achieve this goal the different surface properties resulting from the machining process need to be identified and characterized. An excerpt of surface characteristics typically observed in the presented combination of EDM and the Plasma Electrolytic Conversion process is presented in table 2 and divided into three different categories of magnitude.

Table 2: Studied surface characteristics

Selected characteristics of the surface structure for EDM + PEC		
Category 1: Metallurgical	Category 2: Micro topography	Category 3: Macro topography
Crystalline structure	Pores (0 - 2 μm)	Structures e.g. waviness (10 - 50 μm)
White layer	Cracks	Structures e.g. channels (50 - 1000 μm)
Non-parent material	Surface roughness (0.1 - 10 μm)	

Main emphasis of the on-going research is to identify the influence of these surface properties on the biocompatibility and improve or invent processes to avoid those with negative influences and promote those with beneficial results.

Surface characteristics of the first and second category caused by the machining process can include microstructural and metallurgical changes, for example cracks and pores, white layer and changes in the crystalline structure which contribute to a large extend to the surface integrity. As a result of an unsatisfying surface integrity the fatigue strength of the machined part is reduced, which can lead to an early implant failure. Furthermore, properties in these orders can also be the main cause for an increased risk for irritation of the surrounding tissue. This is especially the case if foreign and toxic material is incorporated in the contact area due to

impurities of the alloy or a contamination resulting from one of the machining processes.

The osseointegration is assumed to be significantly influenced by category 2 and 3 of the surface properties shown in table 2. Different studies have given proof that especially the topography of the surface has a significant influence on the cell response. Implants made of titanium with increased roughness and porous surface structures show significantly improved cell adhesion in quantities and time wise compared to smooth surfaces (Buser et al 1991; Gotfredsen et al. 2000). But up to now there is not enough information to discern which kind of surface structure performs best. Some studies show that pore sizes between 50 and 400 μm are desirable (Clemow et al. 1981; Bobyn et al. 1980) and others demand for pore sizes in the range of 2 - 8 μm (Schupbach et al. 2005). Studies analysing sandblasted samples come to the conclusion that osseointegration is generally better with rougher samples. This paper proposes that cells belonging to different steps of the osteoblast differentiation cascade respond differently to the same surface structure. Therefore, a combination of different micro and macro structures generating a multi-scale directed roughness topography may significantly improve the cell response over the whole osseointegration process. In order to identify the optimum composition of influencing variables, the different categories described in table 2 need to be investigated in detail first.

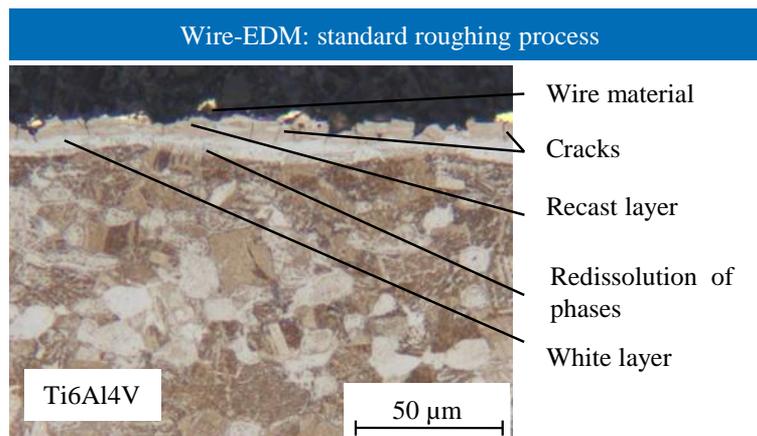
Electro Discharge Machining

Due to the thermal removal principle, any electrically conductive material can be machined by Electro Discharge Machining regardless of its hardness. Therefore EDM is commonly used for difficult to machine materials, e.g. titanium alloys. Since the process is contactless and nearly force free, EDM is best suited for the machining of burr free, complex and filigree 3D-structures with high aspect ratios (Uhlmann 2008).

To analyse the influence of the machining process on the surface properties, a fundamental knowledge of the removal principle is necessary. During EDM small volumes of the machined part are melted and vaporised, due to a sequence of short electric discharges, creating small craters on the surface. These discharges occur between the machined part and a tool electrode which are separated by the working gap filled with a dielectric fluid. The flushing of the fluid removes the debris of the process and leads to a recovery of the dielectric strength in the pulse

interval time between discharges. Since the discharges usually take place at the shortest distance the negative form of the electrode is replicated in the workpiece. The generated craters form the surface of the machined part and their size and shape determine the surface roughness. The form of the craters is directly influenced by the total amount of electrical energy as well as the duration and characteristic pulse form of the discharges. Besides the surface roughness the generator parameters in combination with the machining control also determine the material removal rate, profile accuracy and surface integrity of the machined part. Similar to the crater shape the induced electrical energy is proportional to the material which is removed in each discharge. Therefore a high material removal rate will also lead to an increased surface roughness. But a high material removal rate is also the result of a large thermal impact into the surface area resulting in a broad thermally influenced zone, causing different surface changes. In this heat affected zone metallurgical changes can be observed in form of a white layer, change in crystalline structure and a change in chemical composition, as mentioned in category 1 of table 2. The surface changes observed after a typical rough cut of a Ti6Al4V sample are exemplarily demonstrated in figure 1.

Figure 1: Surface properties of a standard Wire-EDM roughing cut in Ti6Al4V (Klocke et al. 2012)



The literature suggests that these metallurgical and microstructural changes in the top surface layer have a significant impact on the fatigue strength as well as the corrosion resistance of the machined part. These parameters are therefore very important for the machining of implant material, as they directly influence the longevity of the implant. Furthermore, it can be assumed that corrosion of non-biodegradable

implants can lead to irritation of the adjacent tissue. In the case of biodegradable implants the metallurgical changes may also have an impact on the corrosion rate (Denkena and Lucas 2007).

This paper focuses firstly on category 2 and 3 of table 2 as the influence on the osseointegration is expected to be more dominant and these properties are easier to adapt. Characteristics of the surface micro topography are cracks, non-parent material and porosity. Especially unwanted pollution of non-parent material needs to be prevented. In EDM pollution of foreign material usually originates either from the used dielectric or the used electrode. This is the case since not only material of the workpiece is melted but also from the electrode, which can solidify on the workpiece surface. Crack products of the dielectric can also be incorporated into the surface in a similar fashion. Typically deionized water is applied in Wire-EDM and oil based fluids are used in Sinking-EDM. Obviously water can be identified as supposedly biocompatible, whereas the influence of oil based dielectrics need to be thoroughly investigated. Therefore experiments were firstly performed on a Wire-EDM machine tool (Sodick 537L) using a water based dielectric. As electrode material a standard brass wire was applied. Due to the chemical composition of the electrode a contamination of copper is possible and as copper is presumed to negatively affect adjacent tissue, it needs to be monitored.

Table 3: Parameter settings for the analysis of surfaces generated by Wire-EDM

		Parameter											
		ON	OFF	IP	MAO	SV	V	SF	PIK	WK	WT	WS	WP
Number of cut	Main cut	11	15	2215	260	35	8	250	0	25	160	98	45
	1 st trim cut	1	23	2215	750	48	8	6120	0	25	160	98	12
	2 nd trim cut	0	1	1015	0	30	5	7070	8	25	160	98	12
	3 rd trim cut	0	1	1015	0	18	2	7080	9	25	160	98	12

Parameter description*

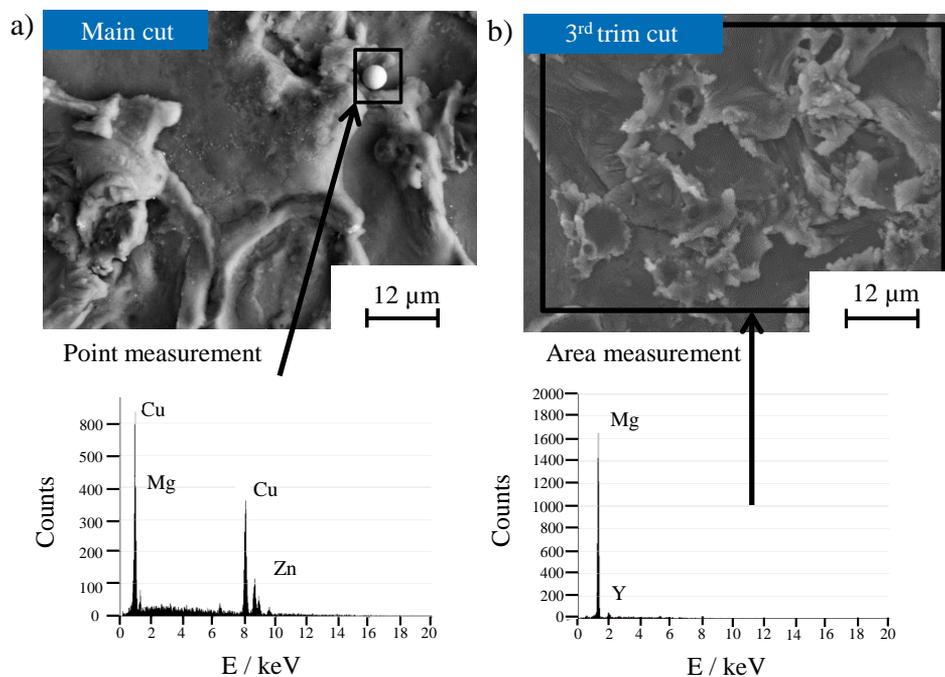
On:	On-time	SF:	Axis speed and control
Off:	Off-time	V:	Open circuit voltage
IP:	Current and generator stage	PIK:	Finishing generator stage
MAO:	Process control	WK:	Wire diameter
SV:	Reference mean voltage	WT:	Wire tension
WS:	Wire speed	WP:	Water pressure

*Process parameters are Sodick specific and do not necessarily resemble a physical quantity.

To prevent negative influences on biocompatibility, adequate parameters for the machining of implants need to be developed. Since the thickness of the thermally influenced zone as well as the surface roughness is a function of the electrical energy which is induced in every discharge, cuts with minimal energies achieve best surface finishes and reduced metallurgical and microstructural (category 1 and 2) changes in the top surface layer. Since the material removal rate is also minimized in this case, not one but a series of cuts (trim cuts) with subsequently reduced discharge energies are performed after the rough cut in Wire-EDM for an effective process.

To identify possible pollution of wire material on the machined surface, several test cuts were performed in WE43 using a brass wire electrode. An excerpt of relevant technology parameters is listed in table 3.

Figure 2: EDX-analysis of WE43 test sample after main (a) and 3rd trim cut (b)



To identify foreign material SEM-images of the surface of every cut were taken and the composition was identified by an EDX-analysis. The surface after the rough cut as well as after the third trim cut is shown in figure 2. Particles on the surface of the rough cut were identified as copper and tin proving that these particles origin from the used brass wire-electrode. The

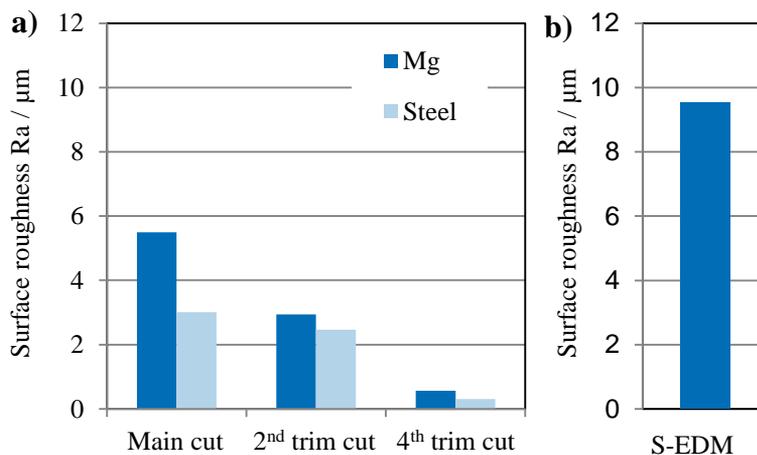
analysis of test samples after the 3rd trim cut on the other hand showed no signs of non-parent material on the surface. An EDX-analysis of the whole area depicted in the SEM-image showed that only magnesium and yttrium which are part of the composition of WE43 as described in table 4 could be identified. As a result it can be assumed that the biological compatibility is not significantly decreased by the EDM process.

Table 4: Composition of the investigated titanium and magnesium alloys

Material	Composition			
WE43	Yttrium 3.7 - 4.3%	Rare earth 2.4 - 4.4%	Zirconium 0.4%	Balance Mg
TiAl6V4	Aluminium 5.5 - 6.7%	Vanadium 3.5 - 4.5%		Balance Ti

The resulting surface roughness of the machined part is assigned to the second and third category of surface properties identified in table 2 and therefore has a great impact on the osseointegration as stated before. By adjusting the generator parameters of the EDM process the roughness can be adapted with high flexibility and with the same electrode. The surface roughness up to the 4th trim cut is shown in figure 3a.

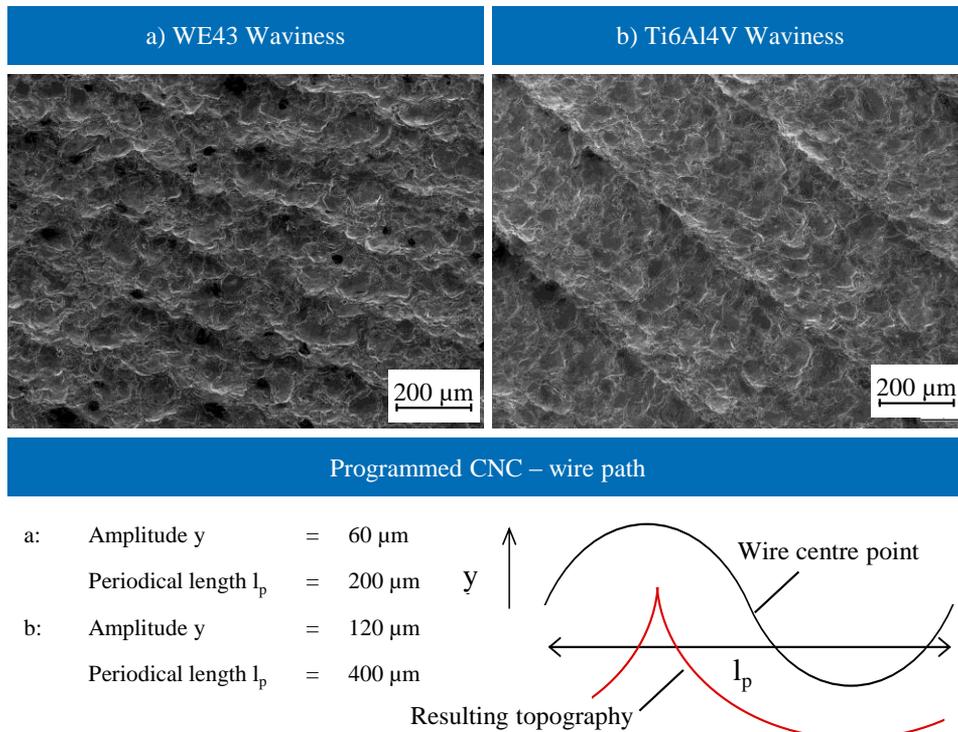
Figure 3: Achieved surface roughness Ra for main- and trim cuts using Wire-EDM, compared to a roughing technology during Sinking-EDM



Using Wire-EDM the surface roughness can typically be adjusted between $Ra = 0.1$ and $Ra = 7 \mu\text{m}$. To prevent the deposition of foreign material also during the main cut it is possible to reduce the overall material which is removed by increasing the gap distance intentionally. This can be done

for example by performing a main cut after the trim cuts with increased offsets. Therefore it is possible to prevent negative influences on the biocompatibility throughout the possible roughness range. As shown in figure 3b using Sinking-EDM an even larger roughness can be obtained. With special technology developments it can be assumed that even higher Ra values could be achieved.

Figure 4: A surface machined by EDM with an overlaid waviness achieved by an approximated sinusoidal cnc wire path



To machine structured surfaces in the magnitude between 10 and 50 μm by Wire-EDM an oriented surface, e.g. a regular waviness as seen in figure 4, can be generated in order to improve the overall topography. In this case a sinusoidal cnc wire path was approximated for the tool centre point with an amplitude $y = 60 \mu\text{m}$ and a periodical length $l_p = 200 \mu\text{m}$ in figure 4a compared to an amplitude $y = 120 \mu\text{m}$ and a periodical length of $l_p = 400 \mu\text{m}$ shown in figure 4b. Since the diameter of the used wire-electrode is 250 μm the produced profile does not have the exact programmed form, but meets the waviness in the amplitude that was intended. In the SEM images it can be seen that this already is an overlap

of a surface roughness and waviness resulting in two adjustable surface structures of different magnitudes.

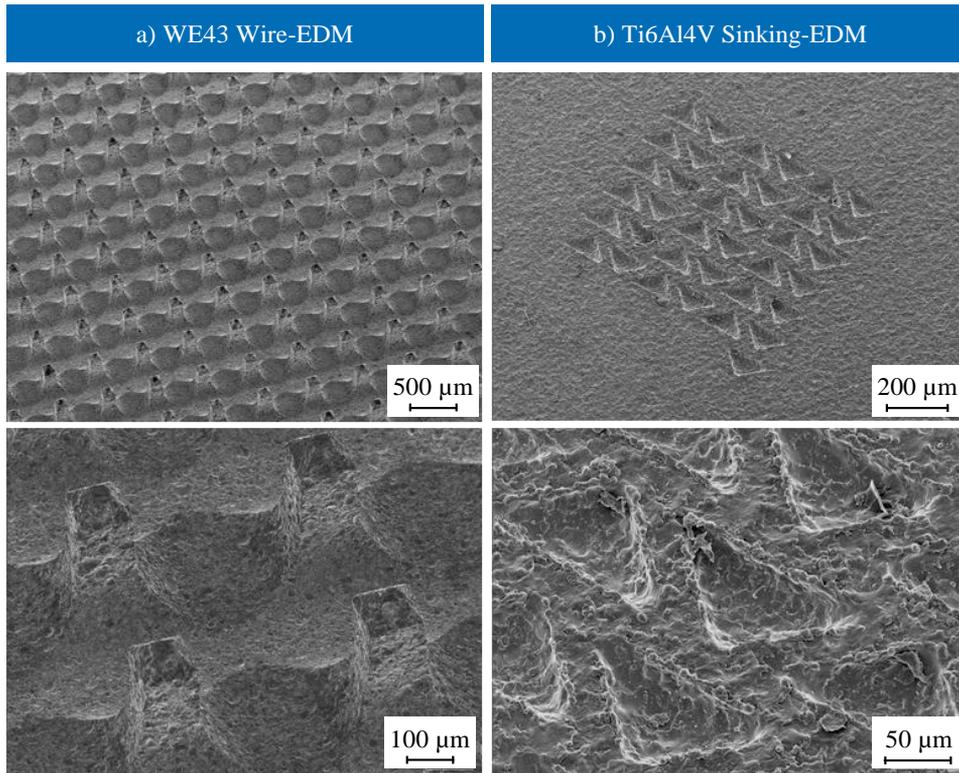
In a similar fashion small and filigree micro-structures also showing an integrated adaptable surface roughness can be created. Figure 5a shows an example of a protruding microstructure machined by Wire-EDM. In this case the smallest possible space in between the regular structures is only restricted by the wire diameter, which was again 250 μm , and the gap size. The width of the rising structure can easily be machined down to 50 μm with standard technologies on a macro Wire-EDM machine. To create the symmetrical structure, the machining process was performed at an angle of 0° and redone at an angle of 90° .

The connection between tissue and implant should be significantly increased especially due to a major increase of the surface area. In this case the embossed structure could also lead to an interlocking connection between implant and bone tissue in order to optimize the seat of the implant. Height, diameter and general form, e.g. rectangle or pyramidal, can be adapted according to future in-vivo and in-vitro testing. Even undercuts are possible. If the dimensions of these regular structures need to be even smaller it is possible to switch to a micro Wire-EDM machine with wire diameters down to 20 μm .

In contrast to Wire-EDM it is possible to exchange the restrictions of object dimension and spacing by applying Sinking-EDM. With an electrode prepared by Wire-EDM regular impressed structures with small dimensions can be generated. In this case the remaining protruding structure in between the objects can easily be machined down to 50 μm , as seen in figure 5b.

In the magnification the overlay of the crater topography and structure becomes obvious, demonstrating the possibility to combine different variable structural properties in different magnitudes.

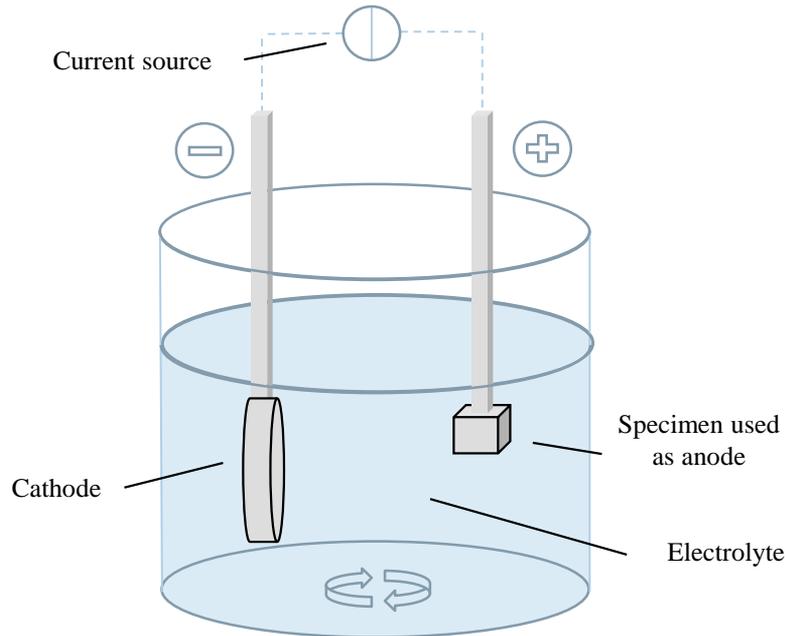
Figure 5: SEM images of regular protruding microstructures (a) in WE43 by Wire-EDM and (b) impressed in Ti6Al4V by Sinking-EDM



Plasma Electrolytic Conversion

The Plasma Electrolytic Conversion of the surface is a highly promising process to enhance the mechanical and chemical surface properties of light metal materials. The process takes advantage of a special material property of certain light metals, e.g. titanium, aluminum, zirconium, or magnesium to form a barrier layer consisting of different types of oxides when exposed to the nature atmosphere. This capability to chemically react with oxygen is technically amplified by subjecting the material to a Plasma Electrolytic Conversion in an aqueous solution. Due to the catalytic partial oxidation running on the material interface, the technically feasible coating thickness of the oxide layer is highly augmented. The schematic set-up for the applied electrochemical process used for this study is shown in figure 6.

Figure 6: Schematic drawing of the Plasma Electrolytic Conversion test setup



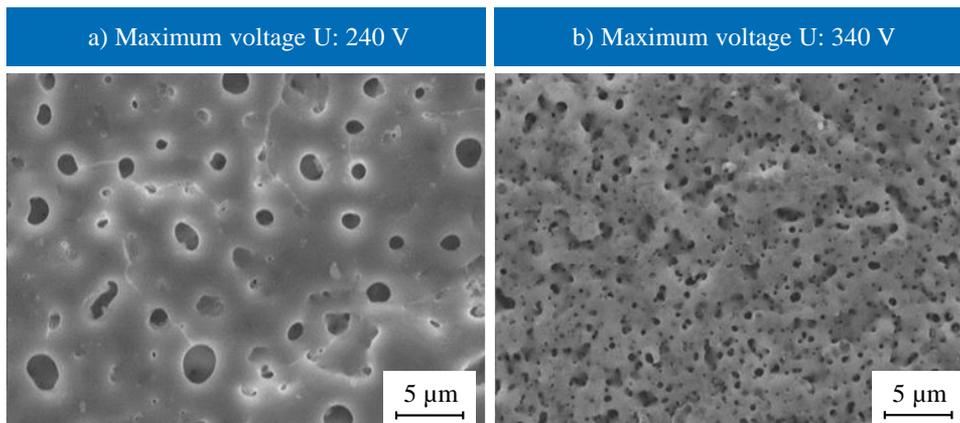
In this case the light material specimen is used as anode. To prevent stray currents all electric contacts need to be made of a barrier layer forming material as well. In order to enable the needed current flow, the system is counter contacted by a cathode in the form of a preferably, circular plate made of stainless steel. After insertion into the electrolyte, the specimen forms a natural oxide layer by a mere chemical reaction in the magnitude of 1 μm . This conversion layer is chemically inert, has a high electrical resistance and prevents further natural chemical reactions. To amplify this layer and augment its properties, a sufficient electrical voltage is applied causing local layer breakdowns in form of discharges between the base material and the electrolyte. These discharges cause local plasma channels which enable mass transport and diffusion processes. Consequently a current flow is enabled leading to a further layer growth. This process continues until the thickness of the layer has grown to the point where the applied voltage is not sufficient anymore to overcome the simultaneously increased electrical resistance. Hence, the voltage is increased until the desired layer thickness is reached. To achieve uniform layer properties the voltage is modified in such a way that the current is held constant throughout the whole process.

The so-created surface layer features different attributes that foster its application for medical use, especially for implants. Due to the electro-

chemical character, the depicted process has to be regarded as a conversion of the surface rather than a typical deposition which is common for ordinary coatings. As a result the layer thickness is distributed uniformly, no shadowing effects take place and the adhesion is very high. By reacting with free oxygen of the aqueous solution, the light material forms an oxide layer showing typical ceramic properties, e.g. high corrosion resistance, enhanced hardness as well as the resistance to wear and abrasion. Therefore the naturally good properties of, e.g. titanium implants, can be improved even further. Due to the chemical inertness of the ceramic like surface conversion the overall biocompatibility should increase significantly. In addition to this the irritation of the surrounding tissue, caused by abrasion particles, is reduced due to the improved wear resistance.

Concerning degradable magnesium implants this conversion process could lead to the first applicable implant design by effectively decelerating the corrosion process and regulating the hydrogen release. The metallurgical surface properties in the magnitude of the first category of table 2 are not yet in the scope of this paper, but will be investigated thoroughly in the future. A generic topographical view of electro-chemically passivated surfaces is shown in figure 7.

Figure 7: Different grades of porous structures generated by the Plasma Electrolytic Conversion of the surface with (a) 240 V and (b) 340 V maximum voltage



The characteristic porous structure of two differently treated specimens can be seen. For this experiment the test conditions described in table 5 were applied. By adjustment of the process parameters, i.e. current, voltage, waveform or chemical composition of the electrolyte, the different parameters of the specific structure can be altered, i.e. the size

and density of the pores can be varied. Since the pore diameter is in the range of micrometers they are part of the second category of table 2 and form another modifiable surface property.

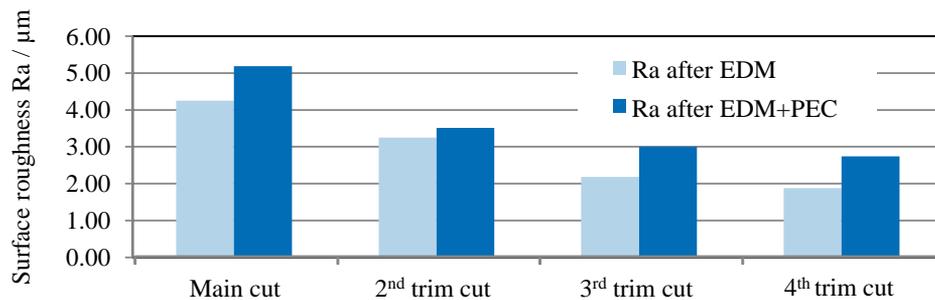
Table 5: Parameters used for the plasma electrolytic conversion process

Electrolyte capacity V / l	2, stirred
Process temperature T / °C	RT – 31, constantly rising
Applied current density J / (A / dm ²)	1.6
Process time t / min	10
Specimen dimensions (w x h x d) / mm ³	20 x 10 x 10
Electrolyte composition	Ammonium dihydrogen phosphate (pure)

Combination of Electro Discharge Machining and Plasma Electrolytic Conversion

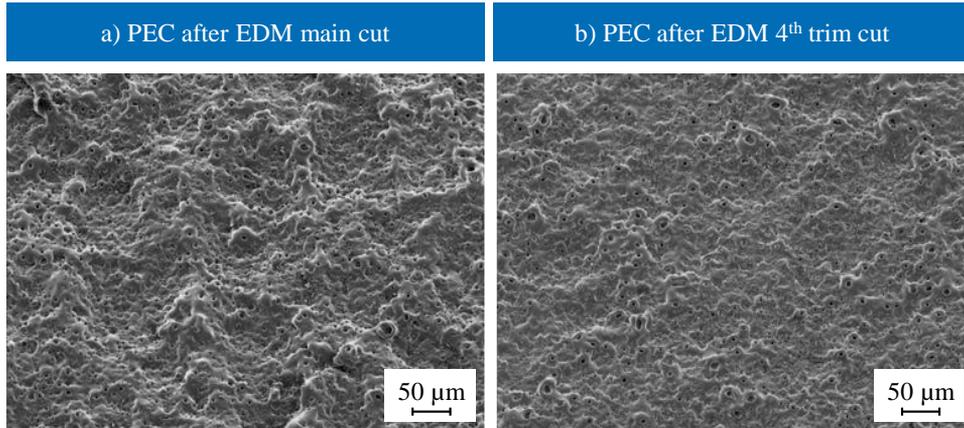
Similar to the combination of oriented macrostructures and surface roughness achieved by the EDM process another dimension can be added to the implant surface by the described subsequent surface transformation. As described before this surface treatment is not a regular coating and therefore no masking of the structures takes place. As a result the initial structure and characteristics of the machined surface remain to a large extent. As shown in figure 8 the resulting surface roughness after the treatment increases but a significant difference between each trim cut can still be observed.

Figure 8: Surface roughness Ra of each cut before and after Plasma Electrolytic Conversion



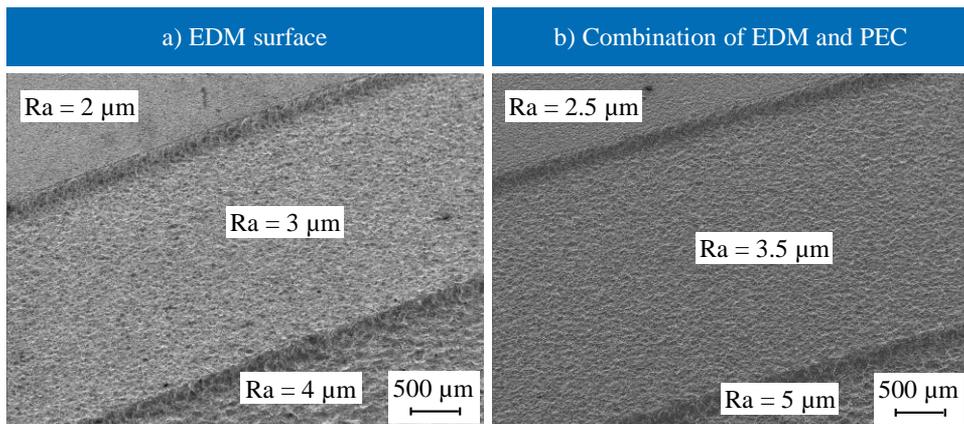
This difference can also be seen in the SEM images which were taken of the surface after the Plasma Electrolytic Conversion, figure 9.

Figure 9: SEM image of the transformed surface of (a) main and (b) 4th trim cut



By this method the variable crater topography of the surface machined by EDM can be combined with a porous structure of pore sizes which can be altered independently. By parameter variation it is even possible to integrate different surface topographies on the desired structures. A simple staircase structure with a different surface roughness on every step machined by EDM is shown in figure 9a next to the same test sample after the surface modification in figure 9b.

Figure 10 Multiple surface qualities machined by Wire-EDM (a) and the same surface after the plasma electrolytic conversion (b)



In this case the same pore size and thickness of the modification layer was integrated on different surface topographies which result in three different surfaces. It is assumed, that the cell response of these surfaces is significantly different. As a result many properties of the surfaces can be

changed independently to increase the biocompatibility of the machined part. In addition to this, different areas of a future implant can easily be equipped with different properties which are especially desirable for implants in contact with different sorts of tissues. These could be dental implants which are in contact with the hard tissue of the jaw and soft tissue of the gums for example.

Conclusion

By the combination of Electro Discharge Machining and Plasma Electrolytic Conversion different surface properties can be adapted independently to functionalize an implant surface on multiple layers of magnitudes, creating a multi-scale directed surface topography. Due to the removal principle of EDM macro geometries demanded by the medical sector like grooves or channels in the magnitude of 50-1000 μm can be created with high aspect ratios as well as a surface roughness in the magnitude of 0.1-10 μm and also a combination of both. In addition to this a regular directed surface structure (e.g. waviness) can be added to improve the surface roughness or to realize structured surfaces in the magnitude of 10 - 50 μm . Using Plasma Electrolytic Conversion a corrosion and wear resistant ceramic layer can be added to the structured surface. This surface modification has superior adhesion properties with no shadowing effects. Therefore the structure beneath this porous layer is preserved to a large amount. Thickness of the layer as well as the pore size is adaptable resulting in a further increase of tailored properties. These properties together with the metallurgical changes, observed in both processes, need to be tested in detail using in-vitro and in-vivo analysis. The results will then be used in order to identify the optimum of each variation which will improve the overall implant behavior.

Acknowledgments

This work was funded by the Federal Ministry of Education and Research in the KMU innovative project BIOMAGIK.

References

- Bobyn, J.D., Pilliar, R.M., Cameron, H.U. and Weatherly, G.C. (1980), 'The Optimum Pore Size for the Fixation of Porous-Surfaced Metal Implants by the Ingrowth of Bone, Clin Orthop Relat Res, Vol. 150, pp. 263-70.
- Buser, D., Schenk, R., Steinemann S., Fiorellini, J., Fox, C. and Stich, H. (1991), 'Influence of surface characteristics on bone integration of titanium implants. A histomorphometric study in miniature pigs', Journal of Biomedical Materials Research, Vol. 25, pp. 889-902.

- Clemow, A.J.T., Weinstein, A.M., Klawitter, J.J., Koeneman, J. and Anderson, J.A. (1981), 'Interface mechanics of porous titanium implants', *Journal of Biomedical Materials Research*, Vol. 15, pp. 73-82.
- Denkena, B. and Lucas, A. (2007), 'Biocompatible Magnesium Alloys as Absorbable Implant Materials –Adjusted Surface and Subsurface Properties by Machining Processes', *Annals of the Cirp*, Vol. 56, pp. 113-116.
- Gotfredsen, K., Berglundh, T. and Lindhe J. (2000), 'Anchorage of titanium implants with different surface characteristics: an experimental study in rabbits', *Clinical Implant Dentistry and Related Research*, Vol. 2, pp. 120-128.
- Gu, X. and Zheng, Y (2010), 'A review on magnesium alloys as biodegradable materials', *Front. Mater. Sci. China*, Vol. 4, pp. 111-115.
- Medical Manufacturing (2009), *Society of Manufacturing Engineers*, pp. 13-19.
- Nasab, M.B. and Hassan, M. R. (2009), 'Metallic Biomaterials of Knee and Hip – A Review', *Trends Biomater. Artif. Organs*, Vol. 24, pp. 69-82.
- Kiesewetter, K., Schwartz, Z., Dean, D.D. and Boyan, B.D. (1996), 'The Role Of Implant Surface Characteristics in the healing od bone', *Crit. Rev. Oral Biol. Med*, Vol. 4, pp. 329-345.
- Klocke, F., Welling, D. and Dieckmann, J. (2012), 'Titanium parts for medical sector by wire-Edm', *Proceedings of the 1st international conference on design and processes for medical devices*, pp. 163-167
- Niinomi, M. (2002), 'Recent Metallic Materials for Biomedical Applications', *Metallurgical and Materials Transactions*, Vol. 33A, pp. 477-485.
- Peters M. and Leyens C. (2002), 'Titan und Titanlegierungen', *Wiley-VCH Verlag*, Vol. 3.
- Shupbach, P., Glauser, R., Antonio, R., Martignoni, M., Sennerby, L., Lundgren, A. and Gottlow, J. (2005), 'The Human Bone-Oxidized Titanium Implant Interface: A Light Microscopic, Scanning Electron Microscopic, Back-Scatter Scanning Electron Microscopic, and Energy-Dispersive X-Ray Study', *Clin Implant Dent Rel Res*, Vol 7, pp. 36-43.
- Staiger, M.P., Pietak, A.M., Huadamaí, J. and Dias, G. (2006), 'Magnesium and its alloys as orthopedic biomaterials: A review', *Biomaterials*, pp. 1728-1734
- Uhlmann, E., Piltz, S. and Oberschmidt, D. (2008), 'Machining of micro rotational parts by wire electrical discharge grinding', *Production Engineering, German Academic Society for Production Engineering (WGP)*, Volume 3, pp. 227-233.
- Williams, D. F. (2008), 'On the mechanism of biocompatibility', *Biomaterials*, Vol. 29, pp. 2941–2953.
- Witte, F., Hort, N., Vogt, C., Cohen, S., Kainer, K.U., Willumeit, R. and Feyerabend, F. (2009), 'Degradable Biomaterials based on Magnesium Corrosion', *Current Opinion in Solid State and Materials Science*